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DIMORVAN BORDIN

INFLUÊNCIA DO MÉTODO DE DEPOSIÇÃO DO DLC NAS PROPRIEDADES MECÂNICAS E PROBABILIDADE DE SOBREVIVÊNCIA DE PARAFUSOS DE PILARES

THE INFLUENCE OF DLC-COATING DEPOSITION METHOD ON THE MECHANICAL PROPERTIES AND RELIABILITY OF ABUTMENT'S SCREWS

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Tese apresentada à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para a obtenção do título de Doutor em Clínica odontológica, na Área de Prótese Dental.

Thesis presentented to the Piracicaba Dental School of the University of Campinas in partial fulfillment of the requirementes for the degree of Doctor in Clinical Dentistry, in Prosthodontic area

Orientadora: Prof. Dra Altair Antoninha Del Bel Cury

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A Ata da defesa com as respectivas assinaturas dos membros encontra-se no processo de vida acadêmica do aluno.

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RESUMO

As complicações protéticas em reabilitações unitárias implantossuportadas envolvem o comprometimento da integridade do sistema de parafuso-pilar onde o afrouxamento e/ou fratura do parafuso do pilar é a falha protética mais frequente. Dessa forma, a utilização de tratamentos de superfície que melhorem o desempenho mecânico dos parafusos pode contribuir para o aumento do sucesso das reabilitações em longo prazo. O objetivo desse estudo foi avaliar a influência do método de deposição do DLC (*Diamond-like carbon*) em parafusos de pilares quanto ao desempenho mecânico de restaurações unitárias implantossuportadas. Para isso, setenta e cinco parafusos de geometria idêntica foram randomizados em três grupos de acordo com o método de aplicação do DLC: CTRL: controle (sem nenhum tratamento); RFPA (DLC aplicado pelo método de *radio frequence plasma-activated*) (grupo experimental); UMS- DLC aplicado pelo método de *unbalanced magnetrom sputtering* (grupo experimental). Inicialmente foi realizada a mensuração das propriedades mecânicas das superfícies tratadas ou não, avaliando a nanodureza e o módulo de elasticidade (ME) de doze parafusos (n=4/grupo). As diferenças entre os grupos foram avaliadas por meio do modelo linear misto. Os dados mensurados no teste de nanodureza foram utilizados como parâmetros para alimentar uma análise de elementos finitos (*in silico*) e avaliar a tensão gerada diretamente sobre o DLC e na interface com o titânio. Para isso, foi modelado um bloco (5 x 5 x 10mm) considerado como substrato de titânio. Sobre ele, foi modelada uma camada representativa de tratamento de superfície de 4µm de espessura e uma contraparte representando uma secção de rosca para aplicação de carga. Foram obtidos três modelos virtuais, simulando a presença dos tratamentos de superfície: Ctrl, UMS e RFPA (n=1/grupo). Sobre a contraparte foi aplicada uma carga de 30 N para simular um torque de apertamento de parafusos. As variáveis respostas foram a tensão de cisalhamento (MPa) e deformação (µm) nas duas superfícies: uma em contato com a contraparte e a outra na interface com o substrato. Para o teste de fadiga progressiva acelerada (*in vitro*), os vinte e um parafusos remanescentes de cada grupo foram conectados à pilares personalizados e esses conectados à implantes de conexão hexagonal externa (4,0 x 10mm). Todos os parafusos receberam o mesmo torque de apertamento de 30 Ncm de acordo com as recomendações do fabricante e verificado por meio de torquímetro digital. Em seguida, uma coroa metálica de um incisivo central superior foi cimentada. Três espécimes de cada grupo foram submetidos ao teste uniaxial de compressão até a falha. Os valores médios de falha foram utilizados para a obtenção de três perfis de carga necessários para o teste de fadiga progressiva acelerada. Os perfis nomeados de leve (n=9/grupo), moderado (n=6/grupo) e agressivo (n=3/grupo) se referiram ao aumento progressivo da carga. A curva de probabilidade de Weibull (resistência característica vs. módulo de Weibull) e a probabilidade de sobrevivência foram calculadas considerando a simulação das missões de 50.000 e 100.000 ciclos e cargas de 100, 150 e 200 N. As amostras fraturadas, mais representativas, de cada grupo foram avaliadas em microscopia eletrônica de varredura (MEV) para caracterização fractográfica. De acordo com os resultados obtidos no teste de nanoindentação, ambos grupos recobertos com DLC demonstraram nanodureza superiores ao controle (p<,01). O grupo UMS demonstrou módulo de elasticidade superior ao RFPA e Ctrl, (p=,00). Não houve diferença estatística entre o módulo de elasticidade entre RFPA e Ctrl (p>,05). A análise in *silico* demonstrou que quanto maior o módulo de elasticidade do DLC, maior a tensão de cisalhamento, especialmente na região de interface entre DLC e substrato de titânio. Em relação ao teste mecânico de fadiga, os grupos CTRL e RFPA demonstraram valores de Beta (ß) <1 (ß=0,68 e 0,62, respectivamente), indicando que a fadiga foi atribuída às falhas intrínsecas do material e relacionada a falhas precoces. Entretanto o grupo UMS apresentou ß=1,14, o que associa a falha ao processo de fadiga, (dano acumulado), que tende a ocorrer de forma tardia. Todos os grupos demonstraram elevada probabilidade de sobrevivência em 100N (99%). No entanto, foi observada uma diminuição significativa na probabilidade de sobrevivência em todos os grupos quando a missão de 200N foi avaliada tanto para 50.000 ciclos (Ctrl: 51,46%, UMS: 66,53%; RFPA: 54,75%) quanto para 100.000 ciclos (Ctrl: 41,14%, UMS: 40,66%; RFPA: 39.56%). Não foi observada diferença significativa entre os grupos experimentais e controle, independente do número de ciclos e carga simulada (missões). A fratura do parafuso foi o principal modo de falha para todos os grupos. Com base nos resultados conclui-se que ambos tratamentos de superfície com DLC aumentaram a dureza de superfície, porém esta não influenciou a probabilidade de sobrevivência dos parafusos.

Palavras-chave: Análise de elementos finitos; Desenho de prótese; Implantação dentária;

ABSTRACT

The prosthetic complications usually involve the abutment-screw integrity, where the abutment's screw is the most affected component. Thus, the study of surface treatments in order to improve the material's mechanical behavior might contribute to increase the long-term success follow-up. The aim of this study was to assess the influence DLC (Diamond-like Carbon) deposition method at the abutment's screw on the mechanical behavior of single implant-supported restorations. For this, seventy-five abutment's screws were allocated into three groups according to the DLC surface treatment method of deposition (25/group): CTRL: control (no treatment); RFPA (Radio Frequency Plasma-activated) (experimental group); UMS (Unbalanced Magnetron Sputtering) (experimental group); First, twelve screws (n=4/group) were characterized by nanoindentation testing, where the nanohardness and Young's modulus were measured by nanoindentation testing at the coated and not-coated groups. Data were statistically evaluated through linear mixed models. The Young's modulus previously measured were used to conduct a finite element analysis (*in silico*). Then, a square block (5 x 5 x 10mm) was constructed to simulate a titanium substrate; Onto the block, a 4 µm-thick surface coating and a counterpart were modeled. Three groups were obtained according to the DLC Young's modulus properties: CTRL (control - no DLC coating), UMS and RFPA. A 30 N-load was applied into the counterpart based on a screw thread cross-section to simulate the screw tightening. The shear stress (MPa) and deformation (µm) were measured considering two locations: loading face and interface between substrate and coating. For the step-stress accelerate life-testing (*in vitro*), twenty-one screws were connected into customized abutments and then into external hexagon implants (4.0 x 10mm). All screws were tightening with the same tightening force (30N) using a digital wrench and following the manufacture's instruction. Three samples of each group were undergone to the uniaxial compression testing until failure. The mean load-to-fracture were used to design the step profiles as followed: mild (n=9/group), moderate (n=6/group) and aggressive (n=3/group) according to the speed rapidness increase. The use of level probability Weibull curves and reliability for a given mission of 50,000 and 100,000 cycles at 100 N, 150 and 200N were calculated. The most representative fractured samples were observed under scanning electron microscopy (SEM) to fractography characterization. According to the nanoindentation results, both DLC groups showed higher nanohardness than control ($p<.01$). The UMS showed higher elastic modulus than Ctrl and RFPA ($p=.00$). There was no difference between the elastic modulus of CTRL and RFPA ($p>.05$). The *in silico* analysis showed that, the higher the Young's modulus the greater the shear stress at interface between substrate and coating. Regarding the fatigue testing, the CTRL and RFPA showed ß<1, (ß =0.68 and ß=0.62, respectively), indicating that failures were attributed to materials strength (egregious flaws) associated to early failures. Nevertheless, the UMS showed ß =1.14 indicating that fatigue contributed to accelerate failure (damage accumulation) and tends to behave as late failures. All groups showed high reliability at 100 N-mission (99%). However, a decreased reliability was observed at 200 N for all groups at 50,000 cycles (Ctrl: 51.46%; UMS: 66.53%; RFPA: 54.75%) and 100,000 cycles (Ctrl: 41.14%; UMS: 40.66%; RFPA: 39.56%). There was no difference between experimental and control group regardless the number of cycles and loads simulated (missions). The screw fracture was the chief failure mode for all groups. According to the results, it can be concluded that both DLC-coatings increase the hardness but did not influence the probability of survival of abutment's screws.

Key-Words: Dental Implants; Prosthesis design; Finite element analysis;

SUMÁRIO

1 INTRODUÇÃO

Os implantes dentários têm se consolidado como uma modalidade de tratamento previsível para a reabilitação parcial ou total de dentes ausentes. Embora essa modalidade de tratamento tenha apresentado previsibilidade com elevadas taxas de sucesso (~94,8% em dez anos)(Park et al., 2017), falhas biológicas e/ou mecânicas podem ocorrer devido à variabilidade de geometrias, tratamentos de superfície e conexões protéticas além da resposta biológica individual de cada paciente(Al Jabbari et al., 2008, Buser et al., 2012, Yeo et al., 2014).

As falhas biológicas se referem às complicações referentes à osseointegração ou infecção periimplantar (Park, Kim et al., 2017), ao passo que a as falhas mecânicas frequentemente envolvem o afrouxamento ou fratura do parafuso do pilar devido ao acúmulo de danos por fadiga (Farina et al., 2014, Kourtis et al., 2017). Estudos reportam uma incidência de 12% de falhas em restaurações unitárias envolvendo o parafuso durante um período de 5 anos (Jung et al., 2008, Jung et al., 2012, Zembic et al., 2014). No entanto, é notável que essas taxas podem ser ainda maiores quando se considera apenas os implantes de conexão hexagonal externa (Balik et al., 2012, Michalakis et al., 2014, Sakamoto et al., 2016), uma vez que, devido à reduzida altura do hexágono (0,7mm) há um comprometimento da estabilidade do sistema quando submetido às forças não axiais, causando o deslocamento do pilar e consequentemente falha (Kourtis, Damanaki et al., 2017).

Embora esse tipo de conexão venha sido preferencialmente substituída por conexões cônicas internas, o hexágono externo foi a primeira conexão introduzida no mercado, propiciando um grande número de implantes instalados que permanecem em função mastigatória e necessitam de constante manutenção. Além disso, a instabilidade do parafuso pode ser responsável pela micromovimentação do conjunto pilar-parafuso, acarretando em um maior braço de alavanca e/ou sobrecarga de tensões no tecido ósseo e consequentemente falha biomecânica (Gratton et al., 2001, Kourtis, Damanaki et al., 2017). Clinicamente, a falha do parafuso em próteses cimentadas pode ser ainda mais crítica uma vez que a remoção do fragmento fraturado seguramente acarretará em no dano da coroa protética(Khraisat et al., 2004, Michalakis et al., 2003).

O afrouxamento e a fratura do parafuso de retenção do pilar estão associados à redução da pré-carga ou à fadiga do parafuso(Basilio Mde et al., 2012, Farina et al., 2014). Durante o apertamento, uma força rotacional é aplicada sobre a cabeça do parafuso fazendo com que o mesmo seja alongado e direcionado para que o pescoço assente na porção interna do pilar (Haack et al., 1995, Lang et al., 2003). A recuperação elástica do parafuso de titânio gera uma força de apertamento denominada de pré-carga, o qual é responsável pela manutenção da estabilidade do conjunto implante-pilar-parafuso durante a mastigação (Farina et al., 2014, Haack et al., 1995). Tem-se reportado que quanto maior a pré-carga, menor a chance do parafuso falhar uma vez que a estabilidade será maior(Farina et al., 2014). No entanto, aproximadamente 10 a 24,9% da pré-carga inicial é perdida devido ao escoamento do titânio conhecido como *creep* (Dixon et al., 1995, Farina et al., 2014, Haack et al., 1995).

De modo a reduzir o *creep* do titânio, existem tratamentos de superfície que podem ser utilizados para melhorar as propriedades mecânicas destes materiais na tentativa de postergar a ocorrência de falhas. O tratamento de superfície conhecido como *Diamond-like carbon* (DLC) é utilizado na forma de recobrimento onde é possível melhorar as características de superfícies dos materiais sem comprometer as propriedades mecânicas como um todo(Corazza et al., 2014). O DLC é uma forma metaestável de carbono amorfo que contém uma porção significativa de fase sp^3 e confere características semelhantes às do diamante. Esse tipo de tratamento é responsável pelo aumento da dureza, resistência à corrosão, diminuição do coeficiente de atrito e biocompatibilidade do material, sendo utilizado nas áreas de ortopedia e medicina cardiovascular (Diez et al., 2012, Grill et al., 1999) . Além disso, o tratamento melhora o acabamento da superfície recobrindo irregularidades provenientes do processo de fabricação(Vercammen et al., 2000).

Em odontologia, esse tratamento tem sido aplicado em parafusos de retenção de pilares de próteses implantossuportadas, o qual atua como um lubrificante sólido e diminui o coeficiente de atrito (Geoffrey Dearnaley et al., 2005, Kim et al., 2005). Com um menor coeficiente de atrito durante o apertamento do parafuso, há uma menor resistência ao alongamento e consequentemente maximização da pré-carga, diminuindo a chance de ocorrer micromovimentações que podem acarretar em afrouxamento ou fratura (Corazza, de Moura Silva et al., 2014).

Diversos estudos têm avaliado a aplicabilidade do DLC com o objetivo de diminuir a incidência de afrouxamento do parafuso em próteses implanto-suportadas(Basilio Mde et al., 2012, Corazza, de Moura Silva et al., 2014, de Moura et al., 2017, Diez, Brigagao et al., 2012, Hirata et al., 2015, Kim, Lee et al., 2005). No entanto, os resultados reportados são contraditórios. A divergência entre os resultados pode ser atribuída aos diferentes métodos de deposição, uma vez que o carbono possui três formas de hibridização: sp^1, sp^2 e sp^3. Quanto maior a quantidade de carbono no formato sp^3, melhores as propriedades mecânicas do recobrimento ("*diamond-like*") e melhor o desempenho (Vercammen, Haefke et al., 2000). Por outro lado, quanto maior o conteúdo sp^2 ou sp^1, maior a similaridade com as propriedades do grafite, sendo essas propriedades indesejáveis para aplicabilidade odontológica (Corazza, de Moura Silva et al., 2014, Geoffrey Dearnaley et al., 2005, Grill et al., 1999).

Diversas metodologias podem ser utilizadas para aplicação do DLC, sendo que o conteúdo de carbono sp^3, bem como as propriedades mecânicas do tratamento são dependentes da metodologia de deposição. Entre os métodos, destacam-se o canhão de íons, o jateamento, arco catódico, laser pulsado, entre outras. Outras duas metodologias utilizadas na área médica denominada *unbalanced magnetrom sputtering* (UMS) o qual consiste na decomposição do material seguida da deposição física de seu vapor (DFV) pelo bombardeamento dos átomos de carbono. Por outro lado, o método denominado *radio-frequence plasma-activated* é um método de deposição química de vapor (DQV), o qual consiste na utilização de gases precursores que reagem e formam carbono na superfície. Ambos métodos podem ser utilizados em odontologia para deposição de carbono visto à versatilidade das metodologias além da possibilidade de rotação da amostra durante a deposição (Robertson. et al., 2002, Scheibe et al., 1997).

Considerando os fatores envolvidos nas falhas descritos bem como, a possibilidade de aperfeiçoamento do conjunto pilar-parafuso, o objetivo desse estudo foi avaliar a influência do método de deposição do DLC (*Diamond-like carbon*) em parafusos de pilares quanto ao desempenho mecânico de restaurações unitárias implantossuportadas.

2 ARTIGO: The effect of DLC-coating deposition methods on the reliability and mechanical properties of abutment's screws.

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Abstract

Objective: To assess the influence of DLC-deposition method on the probability of survival (reliability) and mechanical properties of abutment's screws. in implant-supported restorations.

Methods: Seventy-five abutment screws were allocated into three groups according to the coating method: Control (no coating); UMS – DLC applied through unbalanced magnetron sputtering; RFPA-DLC applied through radio frequency plasma-activated. Twelve screws (n=4) were used to determine the hardness and Young's modulus (YM) by nanoindentation. A 3D finite element model composed of titanium substrate, DLC-layer and a counterpart (screw's thread cross-section) were constructed. The deformation (µm) and shear stress (MPa) were calculated. The remaining screws of each group were torqued into external hexagon abutments and subjected to step-stress accelerated life-testing (SSALT). The probability Weibull curves and reliability (probability survival) were calculated considering the mission of 100, 150 and 200N at 50,000 and 100,000 cycles.

Results: DLC-coated experimental groups evidenced higher hardness than control ($p<.05$). *In silico* analysis depicted that the higher the surface YM, the higher the shear stress. Control and RFPA showed ß<1, indicating that failures were attributed to materials egregious flaws; UMS showed ß>1 indicating that fatigue contributed to failure. High reliability was depicted at a mission of 100N. At 200N a significant decrease in reliability was detected for all groups. No significant difference was observed among groups regardless of mission. Screw fracture was the chief failure mode.

Significance: DLC-coatings did not improve the reliability of implant-supported restorations regardless deposition method..

Key Words: Biomechanics; Fatigue; Reliability; Weibull; Step-stress accelerated life-testing; abutment screw; Screw design.

1. Introduction

When prosthesis service involves sliding between metallic surfaces, as occurs in implant-abutment-screw contact area, severe wear (known as galling) may occur. Under loading conditions, materials with lower modulus of elasticity adheres locally to the contacting opposite surface and forms hard “galls” where wear initiates [1, 2]. Any force that causes slippage between threads might reduce preload, which is supposed to maintain the joint clamped, leading to component failure. [3] Clinical findings have reported screw failure incidences of 8.8% for single and 5.4% for splinted restorations over 5-years follow-up [4]. This complication is highly undesirable as it involves additional clinical appointments and, consequently, increases treatment costs. Moreover, component failures can lead not only to implant failure but also gum pain, inflammation, hyperplasia, fistula, and bone loss [1].

To overcome these failures, Diamond-Like Carbon (DLC) coating has been extensively indicated to coat biomedical components since it improves a wide range of surface goods without compromising their bulk properties [5]. Concerning implant-supported prostheses, the rationale behind DLC-coating indication lies on its biocompatibility, high hardness, wear resistance, and low friction coefficient [6, 7]. Additionally, its smooth surface finishing acts as a protective barrier that prevents surface wear debris and toxic elements release, which can be potentially allergenic, inflammatory or carcinogenic [8].

Nonetheless, the literature seems to be contradictory regarding the performance of DLC-coating when applied to implant dentistry. While studies have reported improved mechanical properties of implant-supported restorations when abutment screws are coated with DLC [6, 9, 10], others have reported no significant difference between coated vs. non-coated screws [7, 11-13].

These inconsistent results rely on film properties, such as adhesion characteristics, hydrogen content and density, which seems to be directly dependent on carbon deposition method [14, 15]. Carbon form has a great variety of crystalline structures due to its three hybridization forms: sp^1, sp^2, and sp^3. As expected, an increased surface hardness and elastic modulus correlate with an increased sp^3 percentage and a decreased hydrogen content since its configuration is similar to diamond. In contrast, an increased sp^2 content provides a similar behavior to pure graphite which is less desired for high-load demanded components [16]. Additionally, mechanical properties rely on substrate temperature during DLC coating and can be commonly improved with increased ion energy per condensed carbon atom according to the deposition method [16].

The unbalanced magnetron sputtering (UMS) is a typical and controllable method to produce hydrogen-free films sputtering pure graphite targets using an Argon plasma source through physical vapor deposition (PVD) [17]. Magnetos are used in order to increase the ionization degree of plasma and increase PVD deposition rate on the substrate [17]. On the other hand, radio-frequency plasma activated (RFPA) is a chemical vapor deposition (CVD) method which involves the decomposition of a selected precursor gas, such as methane, ethane, ethylene, acetylene, etc., at high temperature and low pressure to improve the ion radical fraction. The energy for the chemical reaction is provided by plasma, usually created by radio frequency between two electrodes in a chamber filled with the reacting gases [17, 18]. Both deposition methods are commercially available and can be easily applied into screws design.

Clinical trials are highly desirable to analyze implants mechanical performance, however, due to high-cost and time-consuming involved, *in vitro* investigations are first recommended. Considering the strength degradation of implant-abutment-screw-prosthesis system in function and the fact the DLC-coating enhances material's mechanical properties, the present study conducted a coating mechanical measurement by nanoindentation and also, a finite element analysis to study the stress distribution behavior under loading; additionally, a step-stress accelerated life-testing (SSALT) was performed to evaluate the probability of survival (reliability) and failure mode of DLC-coated screws through different deposition methods (UMS and RFPA).

The postulated hypotheses were: (1) DLC-coated screws would present higher probability of survival than non-coated ones; (2) The DLC deposition method would not influence the mechanical properties of coated screws.

2. Materials and Methods

2.1 Experimental design

Seventy-five abutment's screws were allocated into three groups according to DLC-coating method (n=25/group); Uncoated (Control); DLC applied by Radio frequency plasma-activated method (RFPA); DLC applied by unbalanced magnetron sputtering method (UMS). Twelve screws (n=4/groups) were undergone to the nanoindentation testing to measure the elastic modulus and hardness of coating layer and titanium; A finite element model composed of a DLC-coating layer (UMS or RFPA), a titanium substrate and a counterpart (screw's thread cross-section) were characterized using the elastic modulus measured by nanoindentation testing and the shear stress and deformation were calculated. The remaining sixty-three screws were used for the step-stress accelerated life-testing (n=21/group) (Figure 1).

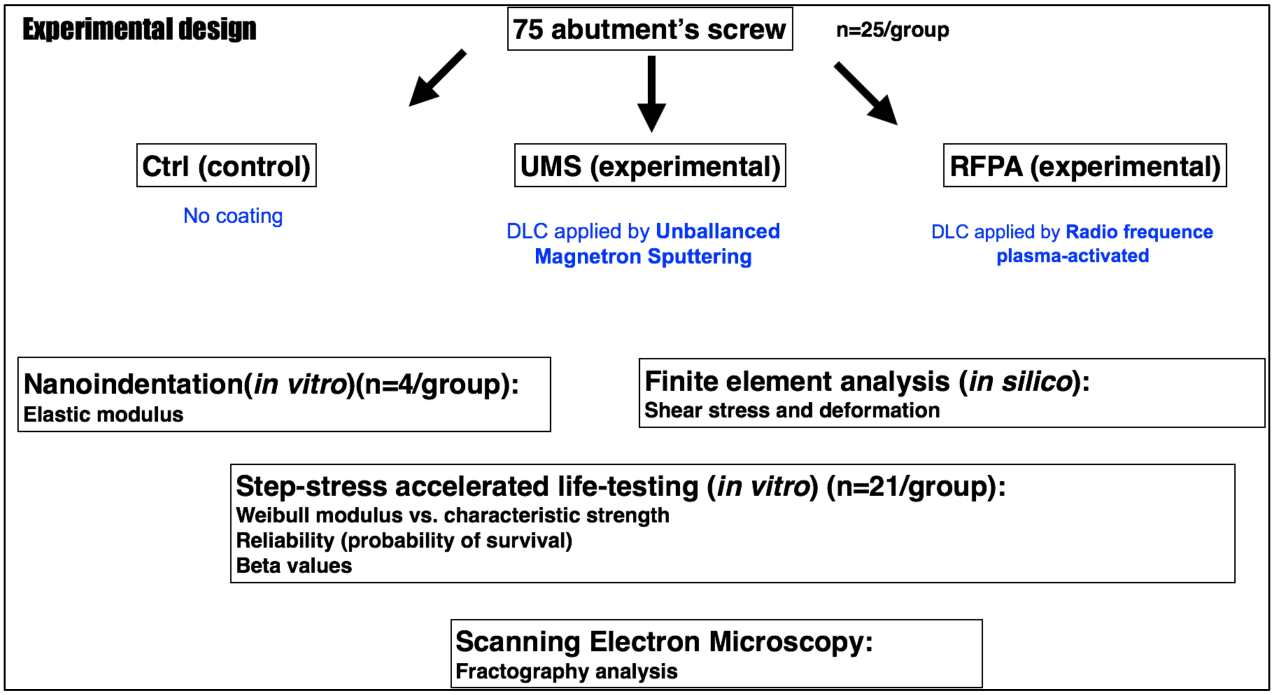


Figure 1 shows the experimental design.

2.2 Nanoindentation testing (*in vitro*)

Twelve abutment's screws (n=4 per group) were embedded into acrylic resin and prepared for nanoindentation testing by grinding (400-1200 grit SiC abrasive papers) (parallel to the longitudinal axis) and polishing (diamond suspension solutions of 9-1 µm particle size) under water irrigation through a grinding machine (Metaserv 3000, Buehler, Lake Bluff, IL, USA).

Nanoindentation was performed using a triboindenter (TI 950, Hysitron, Minneapolis, MN, USA) mounted with a Berkovich diamond three-sided pyramid probe. A loading profile was designed after a pilot test. A peak load of 4000µN was applied, followed by 10 seconds of holding and 3 seconds for unloading the probe. For each specimen, 10 nanoindentations were performed on the coating layer of experimental groups, as well as in the same region of control group (titanium surface).

From each generated load–displacement curve, the reduced modulus E_r (GPa) and hardness H (GPa) of coating layer and titanium were computed via Hysitron TriboScan® software using the following equations 1 and 2, respectively [19]:

$$E_r = \frac{\sqrt{\pi}}{\sqrt{2A(h_c)}} \times S$$

Equation (1)

$$H = \frac{P_{max}}{A\ (h_c)}$$

Equation (2)

Where S is the stiffness, h_c is the contact depth, P_{max} is the maximum applied force (4000 μN), and $A(h_c)$ is the contact area computed from the TriboScan® software utilizing the area function with respect to the contact depth.

Through the reduced modulus E_r, the corresponding elastic modulus E_b (GPa) was calculated using the following formula[19]:

$$\frac{1}{E_r} = \frac{1 - V_b^2}{E_b} + \frac{1 - V_b^2}{E_i}$$

Equation (3)

where v_b (0.35) is the Poisson's ratio for titanium [20], E_i (1140 GPa) is the elastic modulus of the indenter, and v_i (0.07) is the Poisson's ratio for the indenter [21, 22].

The rank hardness and elastic modulus were designated for statistical analysis since preliminary data analysis evidenced deviations from normality. Nanoindentation data are presented as estimated ranked means with corresponding 95% confidence interval values (mean ± 95% CI). Data were collected and aligned along with a linear mixed model with fixed factor of screw surface treatment and a random intercept. The analysis was accomplished using IBM SPSS (SPSS, IBM Corp., Armonk, NY, USA)

2.3 Non-linear Finite Element Analysis (FEA) (*in silico*)

A virtual tribology test was conducted to measure the stress generated on the coating interface. Three tridimensional models were constructed to simulate the DLC-coating applied through different deposition methods: UMS- DLC applied trough unbalanced magnetron sputtering; RFPA- DLC applied through plasma-activated radio-frequency, and; Control-uncoated.

A tridimensional square block model (5 x 5 x10 mm) was constructed to reproduce the titanium substrate. Over the substrate, a 4-μm thick coating-layer was modeled to reproduce the DLC film of the experimental groups. A triangular counterpart was modeled to apply the load and simulate the cross-section of a screw's thread (SolidWorks 2013, Dassault Systèmes, Waltham, MA, USA). A simplified model design was assumed to allow mesh generation in the coating-layer body.

The model was exported to the AnsysWorkbench software (Ansys Inc. Canonsburg, PA, USA) for the mathematical analysis. A tetrahedral-quadratic mesh was obtained using the quality element criteria. Manual refinements were conducted on the regions of interest: coating layer, coating-substrate interface and counterpart-coating/substrate interface. A vertical 30-N load was applied at the top surface of the counterpart as in the screw tightening. Full constrain condition (X, Y, and Z axis) was defined to the substrate block while a displacement restriction was used to the counterpart (X and Z constrain). All models were considered as homogeneous, elastic and non-linearly elastics. No separation contact was assumed between the counterpart and coating. The Young modulus (GPa) obtained from the nanoindentation test was used to mechanical characterization (Table 1). Data were quantitatively evaluated using the maximum and minimum principal, respectively tensile and compression stress and, shear stress.

2.4 Step-stress accelerated-life testing (SSALT) (*in vitro*)

Sixty-three external hexagon dental implants (Ø4.0 x 10mm) (#IHEN-4010, Emfils Colosso®, Itu, SP, Brazil) composed of grade V titanium alloy (Ti-6Al-4V, ASTM F136) were vertically embedded into acrylic resin (Orthodontic Resin, Dentsply®) and PVC tubes (∅25 x 35 mm) with the implant's platform positioned at the same level of acrylic resin. Customized abutments (n=63), (#ELE-4010, Emfils Colosso®, Itú, SP, Brazil) were selected and allocated with implants into three groups according to abutment screw DLC-coating method (n=21/group); Uncoated (Control); DLC applied by Radio frequency plasma-activated method (RFPA); DLC applied by unbalanced magnetron sputtering method (UMS) (Durit®Albergaria-a-Velha, Portugal).

The deposition protocol was not provided by the manufacture. All screws (#PPP-2085 Emfils Colosso®, Itú, SP, Brazil) were tightened using a digital torque wrench (Tohnichi BTG150CN-S®, Tohnichi America) following the manufacturer's instructions (30 N.cm). Standardized maxillary central incisor metallic crowns (n=63, cobalt-chrome metal alloy, Wirobond 280) were milled from a single .STL file and cemented onto the abutments with resin cement (RelyX UniCem, 3M Oral Care, St. Paul, MN, USA) following the manufacture's instruction. All implants, abutments, screws and crowns were composed of identical geometry with differences restricted to screw-coating method.

Three specimens of each group underwent single load-to-failure testing (SLF). A uniaxial compression load was applied 30° off-axis at the incisal edge of the crown using a flat-tungsten tip at a cross-head speed of 1mm/min until failure (TestResources® 800L, Shakopee, MN). The mean load from the SLF was used to design the profiles to the step-stress accelerated life-testing (SSALT). The remaining specimens (n=18 per group) were allocated into mild (n=9), moderate (n=6) and aggressive (n=3) profiles, following the aspect ratio distribution of 3:2:1. The profiles were named based on the gradual increase load-rapidness in which a sample was tested to reach a certain load-level, where samples allocated into the mild profile took longer time (number of cycles) to reach the same load-level than samples allocated into the aggressive profile [23, 24].

The SSALT was carried out with a servo-all-electric system (Servo-All-Electric Systems, TestResources 800L®) in which the indenter contacts the crown and applies the prescribed load lingually at the incisal edge of the crown, 30° off-axis. The test was conducted under water at 9 Hz until failure, which was considered as bending or fracture of the screw, abutment or implant [23, 25].

Based on the step-stress distribution, the use-level probability Weibull curves (probability of failure vs. number of cycles) was calculated using a cumulative damage model and a power law relationship (90% two-side confidence bound) (Alta Pro 7 Software, Reliasoft) with the use stress of 150N.

The reliability (probability of an item survive for a given mission without failure) was calculated considering a mission of 50,000 and 100,000 cycles at 100, 150 and 200 N load. The Beta (β), which describes the failure rate over the time, was obtained. A contour plot considering the Weibull modulus (*m*) vs. characteristic strength (η) was plotted using the final load-to-failure during SSALT.

2.5 Failure modes

All failed specimens were inspected under a polarized light microscope (MZ-APO Stereomicroscope, Leica MicroImaging, Thornwood, NY, USA) and classified by the above-mentioned failure criteria. To identify failure origin and fractographic marks further scanning electron microscopy evaluation (SEM) (S-3500N, Hitachi, Schaumburg, IL, USA) was performed.

3. Results

3.1 Nanoindentation

The measured hardness and elastic modulus used to the *in silico* analysis are exposed on Table 1.

Table 1. Hardness and elastic modulus as a function of mean standard deviation used to mechanical characterization of the *in silico* testing (raw data).

	Titanium	RFPA	UMS
Hardness (GPa)	8.41 ±1.0 A	23.64 ±7.2 A	87.82 ±10.8 A
Elastic Modulus (GPa)	138.86 ±38.9 a	166.37 ±55.1 b	542.73 ±127.3 c

Different uppercase letters mean statistical difference among group's hardness.

Different lowercase letters mean statistical difference among group's elastic modulus.

Significant differences were observed in either hardness or elastic modulus between coated screws and control group (titanium). The UMS method showed the highest hardness, followed by RFPA and control ($p<0.001$). Regarding elastic modulus, UMS group showed significant higher values than RFPA ($p=0.01$) and Control ($p<0.01$). No significant difference was observed between RFPA and Control ($p=0.45$).

3.2 Finite element analysis

Table 2 shows the deformation and shear stress. The UMS coating group demonstrated the highest shear stress at both loading surface (73.9 MPa) and interface (173.11 MPa). The higher the DLC elastic modulus, the lower the deformation, and thus an increased shear stress was observed at both interface and loading surface. Figure 2B shows the qualitative stress distribution on both surfaces.

Table 2-Total deformation (μm) and maximum shear stress (MPa) according to the DLC deposition method (UMS or RFPA) and face (loading face or interface between coating and titanium substrate).

	Titanium	Loading face	Interface

		UMS	RFPA	UMS	RFPA
Deformation (µm)	0.15	0.15	0.17	0.15	0.17
Shear (MPa)	6.81	73.90	24.32	173.11	37.76

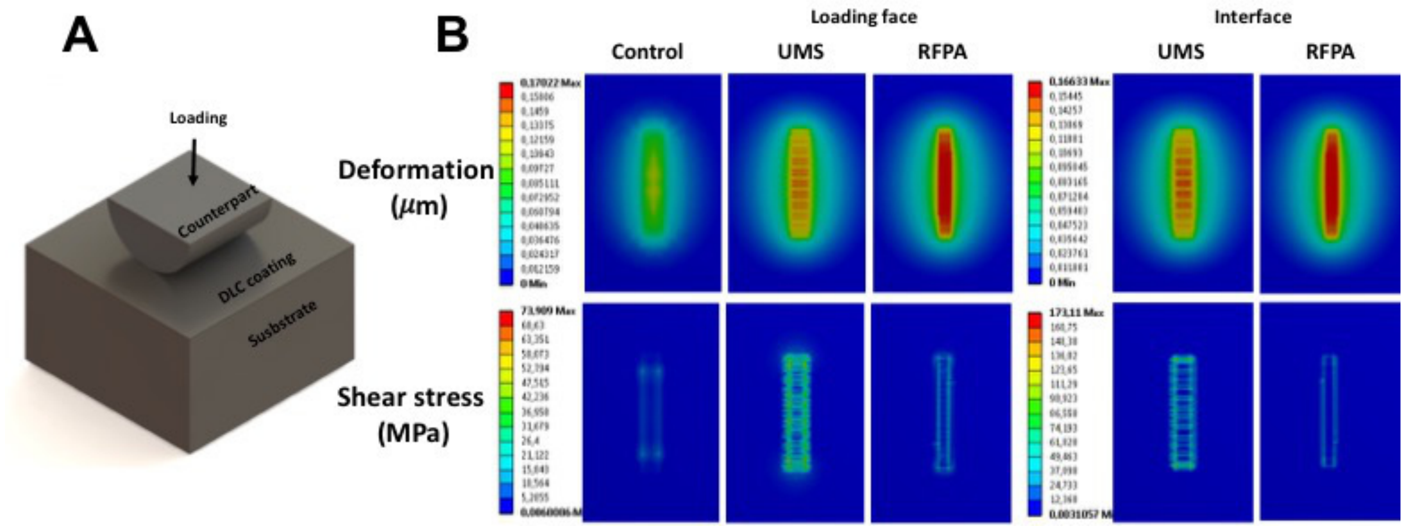
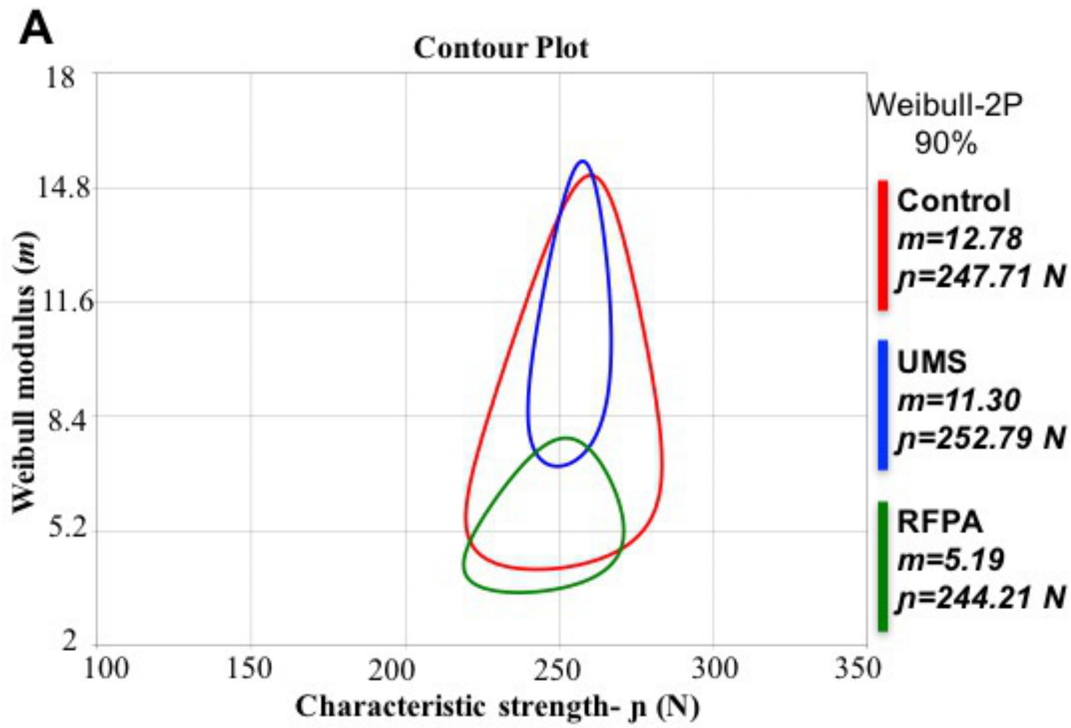


Figure 2A shows the 3D model of the in silico analysis. The DLC-coating layer was standardized as 4μm-thick. Figure 2B shows the qualitative stress distribution at the DLC-coating according to the deposition method (UMS or RFPA) and the face (loading face or interface with titanium substrate).

3.3 SSALT

The mean Beta values (β) derived from the use level probability Weibull was 0.68 for control and 0.62 for RFPA indicating that failures were attributed to materials strength (egregious flaws) rather than fatigue, where failure rate trends to decrease overtime, frequently associated to early failures. Conversely, UMS showed β =1.14 indicating that fatigue contributed to accelerate failures, where failure rate usually tends to increase overtime due to damage accumulation (late failures). [23, 24]

The calculated Weibull modulus (*m*) and characteristic strength (η) are depicted in the contour plot (Figure 3A). Although control group evidenced higher Weibull modulus (*m*=12.78) than UMS (*m*=11.30) and RFPA (*m*=5.19), no significant difference was observed among them considering the overlap of the contour plots. Regarding the characteristic strength (η), no difference was observed among control (η =247.71N), UMS (η =252.79N), and RFPA (η =244.21), also due to overlap between the contours. The use level probability Weibull, probability of failure vs. number of cycles is depicted in figure 3B and shows that probability of failure increases according to the number of cycles.



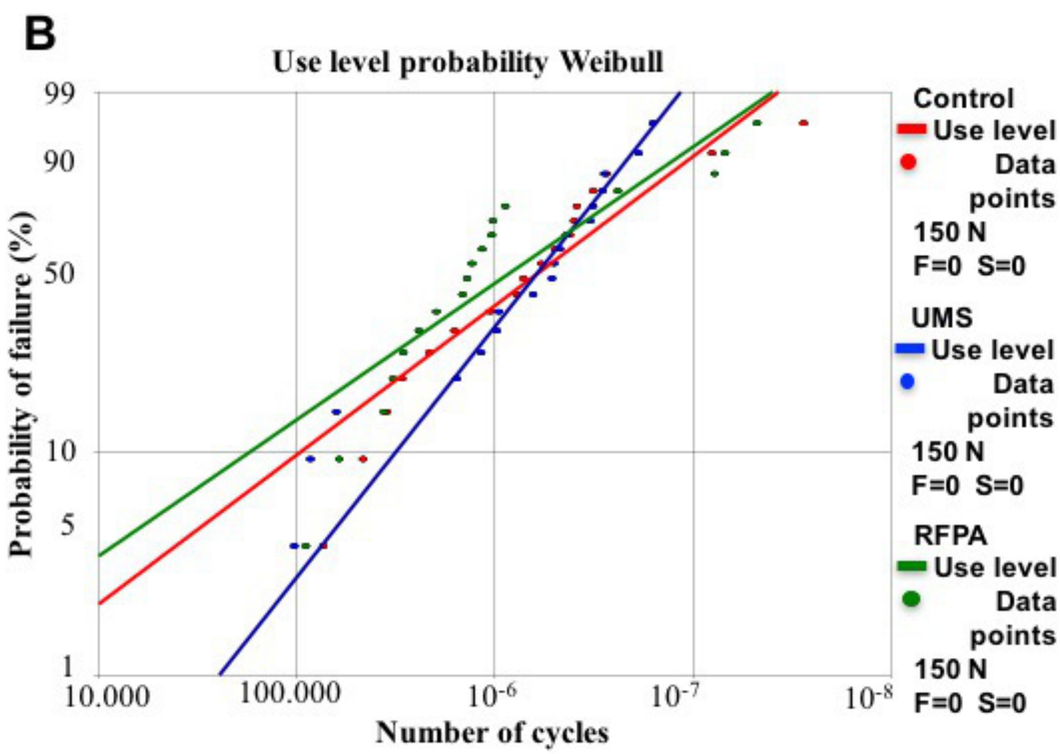


Figure 3A. Contour plot shows the Weibull modulus (m) vs. characteristic strength (η) that represents the load which 63.2% of the samples may fail. Figure 3B. The use level probability Weibull shows the probability of failure (%) vs. number of cycles.

The calculated reliability with 90% confidence interval (CI) for a given mission of 50,000 and 100,000 cycles at 100, 150 and 200N is shown on Table 3. At 100 and 150 N load, all groups showed high reliability (above 86%). When the load was increased to 200 N, all groups showed a significantly decreased reliability (ranging from 66.5% for control group at 50,000 cycles to 39.56% for RFPA at 100,000 cycles), but no significant difference was observed among groups considering the overlap of upper and lower CI bounds.

Table 3. Calculated reliability (%) (probability survival) for a given mission of 50,000 and 100,000 cycles at a load of 100, 150 and 200 N.

		100N			150N			200N		
		Control	**UMS**	**RFPA**	**Control**	**UMS**	**RFPA**	**Control**	**UMS**	**RFPA**
50.000 cycles	Upper bound	99.96%	100%	99.88%	97.97%	99.77%	96.71%	74.87%	82.82%	71.64%
	Reliability	99.70%Aa	99.99%Aa	99.29%Aa	93.86%Aa	98.74%Aa	90.89%Aa	57.46%Ab	66.53%Ab	54.75%Ab
	Lower bound	97.97%	99.76%	95.81%	82.23%	93.32%	76.14%	34.63%	41.44%	33.68%
100.000 cycles	Upper bound	99.93%	100%	99.82%	97.10%	99.49%	95.32%	68.67%	72.81%	63.83%
	Reliability	99.52%Aa	99.98%Aa	98.91%Aa	90.34%Aa	97.23%Aa	86.32%Aa	41.14%Ab	40.66%Ab	39.56%Ab
	Lower bound	96.61%	99.53%	93.40%	70.44%	85.68%	63.67%	12.26%	7.79%	14.73%

Different uppercase letter means difference between groups (at same load mission).

Different lowercase letter means difference between the same group under different loading missions.

3.4 Failure mode

Screw fracture was the chief failure mode for all groups. Figure 4 shows SEM micrographs of the DLC-coating onto the fractured screws after SSALT.

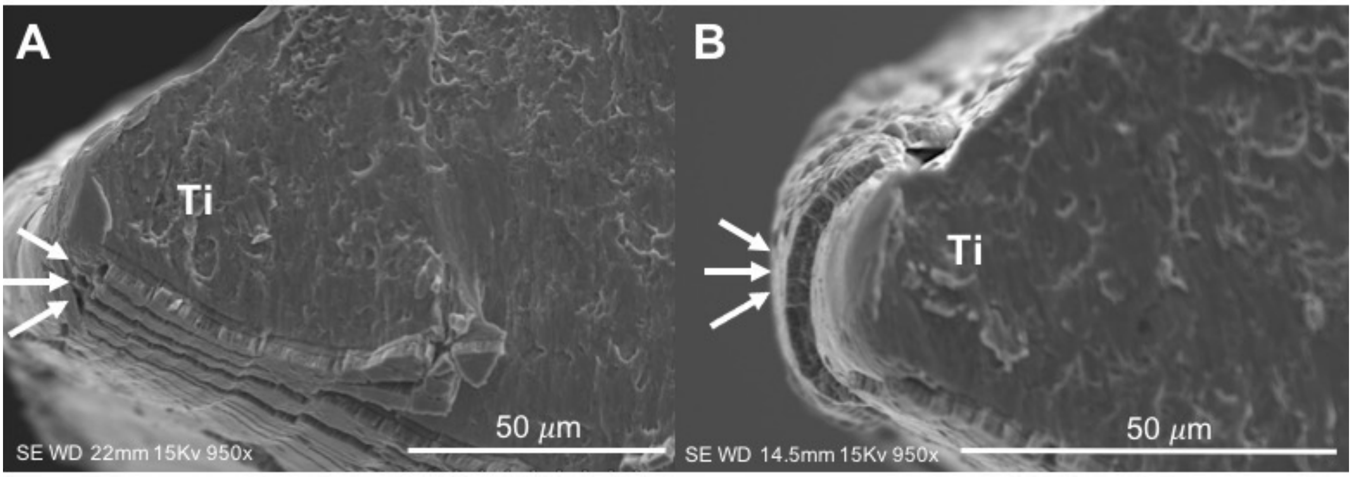


Figure 4 shows fractured screws after SSALT. White arrows indicate the DLC-coating applied under UMS (A) which can be observed a cracked surface; RFPA method (B) applied onto titanium screw's surface (Ti) shows the whole surface.

Figure 5 evidences the fracture origin in the area where tensile stress (lingual surface) was present. Under constant load, a plastic deformation occurred and the initial crack propagated to the opposite direction of the origin, which was subjected to compression stress. A dimple structure represents the end of the fracture, typically observed in the ductile materials failures.

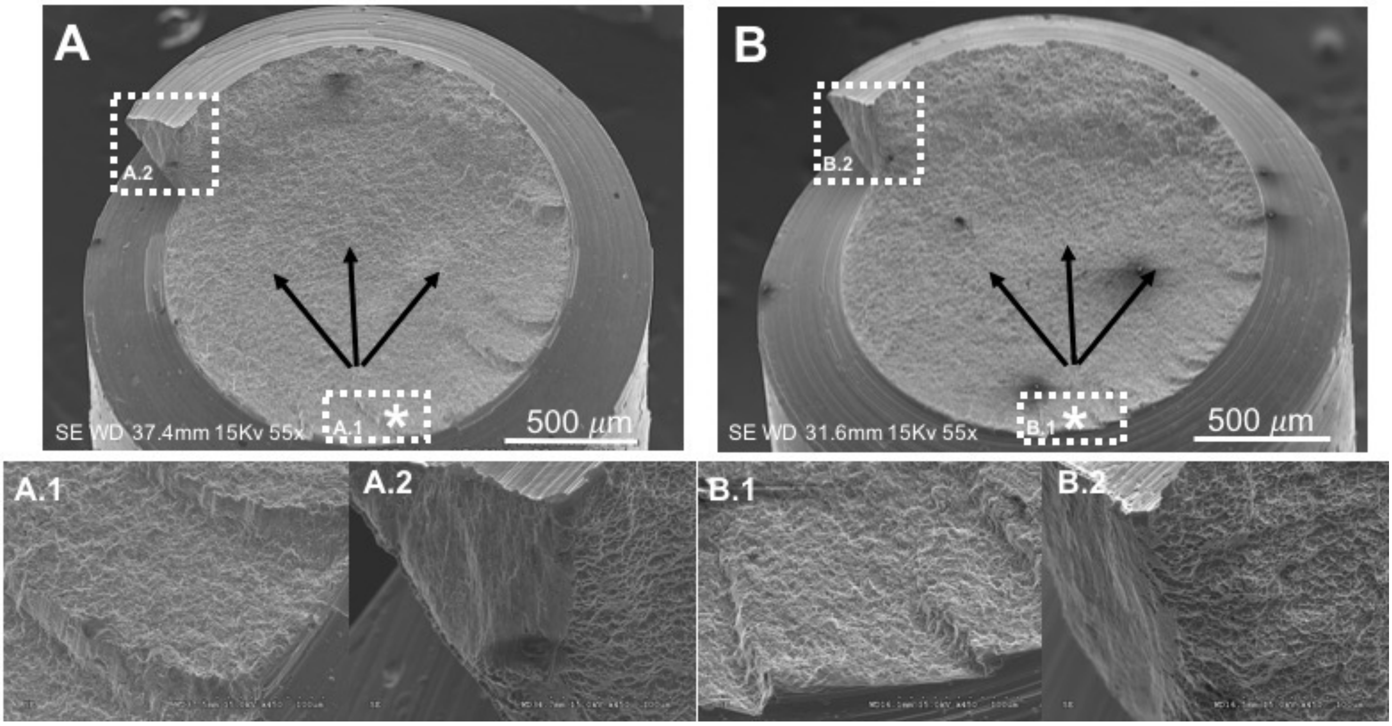


Figure 5 shows the fractured screws after SSALT. A: DLC applied by UMS; B: DLC-coating applied by RFPA method. White asterisk indicates the fracture initiation at the surface underwent to tensile stress. Black arrows indicate the fracture propagation until the opposite surface underwent to compression stress (dimple structure) which represents the end of the fracture.

Discussion

Long-term mechanical stability is highly desired for abutment screws which are frequently challenged by oblique loading commonly resulting in loosening or fracture, especially for single restorations supported by external hexagon connections [26]. The present study evaluated the use of two different deposition methods for DLC-coating to improve implant joint mechanical performance. The first postulated hypothesis that DLC-coated screws would present higher probability of survival than non-coated was rejected since no difference was observed among groups regardless missions. A potential explanation for this result may rely on the observation that DLC coating is used to improve tribologic properties neither improving Ti-6Al-4V bulk properties nor hampering its mechanical properties [27], when used on abutment screws that are a part of a prostheses-implant restored system. Therefore, whereas avoiding galling seems always interesting for abutment screws from a theoretical perspective, DLC-coating methods evaluated herein when tested in screws as part of implant restored crowns, simulating worst case loading scenario, failed to show beneficial or detrimental results. The proposed load missions are relevant considering physiological loads at the anterior region (ranging from 50 to 200 N) [28, 29]. The obtained survival percentage are comparable with the expected behavior of anterior implant-supported rehabilitations and with previous studies using SSALT methodology [25, 27, 30].

When failure rate was assessed, CTRL and RFPA groups showed ß values <1 indicating that failures were dictated by material strength rather than fatigue, usually associated with "early failures". Such failures are usually due to materials' inherent flaws that usually act as flaws precursors that propagates until failure. At a microscopic level, they may act as stress raisers with far-field stress concentrated near their edge. Thus, flaws are associated with a certain stress intensity factor that is related to the far-field stress and the level of stress concentration. When mutual flaw size and stress level lead to a stress intensity factor that surpass the material's fracture toughness, failure occurs [23, 31]. In contrast, UMS group showed ß values >1, indicating that failures were associated with damage accumulation rather than material's egregious flaws, associated with late failures. Usually, strength degradation of titanium and other metals is attributed to the plastic deformation related to their "cold working" process [23].

The second postulated hypothesis that the DLC deposition method would not influence the mechanical properties of coated screws was rejected since experimental groups evidenced higher hardness than control. As previously cited, the key property of DLC coating is dictated by the amount of hydrogen, sp^3-bonded carbon and the cluster structure [17]. The coating is characterized not only by high hardness and high elastic modulus but also by high internal stress, associated to the sp^3-C content. The hardness may range from 10 to 30 GPa with a corresponding Young's modulus 6-10 times higher [14, 32]. Further, the internal compressive stress fluctuates between 0.5 to 6 GPa. If thin coating layers is indicated (less than 1 μm), the film hardness can be even higher (ranging 40-80 GPa) [33] and their Young's modulus can reach up to 900 GPa [34], while the internal stress up to 13 GPa [14]. When incorporating N, Si, O or other metals to the coating layer, a reduction in the internal stresses can be evidenced. However, a decreased hardness and Young's modulus may be experienced as well [35, 36].

The *in silico* findings of this study showed that, the harder the coating layer, the lower the deformation stress (μm). Consequently, an increased shear stress was observed at both interface and loading face of the DLC layer. Under SSALT, the coating was not able to surpass, by itself, the tensile stress generated during loading, thus not altering failure mode between groups neither avoiding facture initiation and propagation.

From a tribological perspective, DLC-coatings are known to form a low shear strength layer of wear debris called as transfer layer, which is responsible for controlling friction phenomenon [15]. The shearing component of the friction accumulated from the energy required to cause a plastic deformation to the transfer layer can vary due to surface conditions [15, 37-39]. The transfer layer is usually composed of carbon wear debris (from the coated surface) as well as the counterface wear debris [15]. This third-body mechanism regulates the frictional reaction by controlling the adhesive interaction between surfaces [15, 37]. Thus, due to DLC-coating effects on controlling friction phenomenon, its indication for coating implant-supported prostheses components has primarily been focused on its potential use to avoid screw loosening [6, 7, 9, 40]. The lower the friction during screw tightening, the higher the preload since a reduced elongation resistance is offered to the screw [3].

Since friction of DLC-coatings can be considered as a summation of adhesive, abrasive and, shear mechanisms [15], the friction-sliding behavior between surfaces with different hardness may represent a problem in a long-term clinical follow-up [2]. The hardest surface tends to wear out the softest one [2], eventually causing irreversible damage on the implant's internal threads. Similarly, relevant wear may occur on the implant hexagon surface when in contact with coated-abutments [41]. Considering the clinical issues regarding implant wear and need for retrieval, future studies involving the DLC application into different components combination (i.e. abutment-screw-implant, implant-screw, etc.) subjected to a wide loading range and wear evaluation between these surfaces are warranted.

4. Conclusion

Although DLC-coating of abutment screws by unbalanced magnetron sputtering or by radio frequency plasma-activated methods increased the hardness compared to uncoated controls, it did not improve the probability of survival of abutment screws used in an implant-supported crown system. Screw fracture was the chief failure mode regardless of DLC deposition method..

Acknowledgements

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3 CONCLUSÃO

De acordo com os resultados obtidos no presente estudo pode-se concluir que o recobrimento do parafuso com DLC (*Diamond-like carbono*) melhora a dureza, porém não aumenta a probabilidade de sobrevivência dos parafusos de pilares.

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